Adrian Bradley

From: Mary P. Goldade
Sent: Friday, August 13, 1999 7:20 AM
To: Adrian Bradley
Subject: FW:

Drexler Comments re: Speciation SOP

-----Original Message-----
From: John W. Drexler [mailto:drexlerj@spot.colorado.edu]
Sent: Thursday, August 12, 1999 3:42 PM
To: Mary P. Goldade
Subject:

Mary:

Perhaps its time to edit the Speciation SOP to make it clear that it can be used for almost any metal and write it a little more generic. It's fine for the pilot study.

Perlite: I don't think that steriomicroscope id is good. Basically the criteria they use to id perlite is glassy (isotropic or nearly so) concoidal fracture and vessicular morphology. Many compounds (amorphous iron, glass, volcanic ash) in nature can give you some if not all of these charcteristics, and soils from the Colorado from range can contain these. I think that EMPA will be helpful. The "perlite" can be counted, sized, and sorted based on chemistry:

Si
Si-Al
Si-Al-Fe
Si-Al-Ca-Fe

In addition, Separate splits for the perlite counting should be made, and mounted on glass thin sections prior to polishing. This will allow one to check each particle counted under polarized transmitted light.

You can get all the data on berlite from www.perlite.org.

John D.

QUESTIONS AND REVISIONS TO METAL SPECIATION METHODS FOR THE VBI70 SOIL CHARACTERIZATION PILOT STUDY

1) Is the method for counting silica sand qualitative or quantitative? If it is a quantitative determination, please summarize the methods that will be used.

The method is quantitative and it is sized and counted just like any element of interest in the speciation SOP. However, again I want to stress that since quartz is the MOST common mineral in the crust its occurrence is meaningless!!!!

2) The following metals have been added to the speciation work:

In	Se
Tl	Sb
Hg	

Table 8.1 and 8.2 (and Table 2.1?) should be revised to include the relevant information about the metal-bearing phase of interest, and the channel/wavelength that will be used for analysis. These tables are attached, for your revision. Please fill in the missing information and fax/email back to me.

DONE

3) Molybdenum is included in Table 8.1 (EMP Standard Operating Conditions). Does this table represent only target metals (e.g., As, Pb, Cd, Zn, In, etc.)? For example, Mo is not a target metal.

Mo was added to the table to remind the operator that with EDS Mo has an overlap with S K alpha and Pb M alpha.

4) We would like at least 200 counts per sample for As;
At least 100 counts per sample for Pb.
Please suggest a similar counting limit for the other metals (Cd, Zn, In, Tl, Hg, Se, Sb), such as a counting or time limit (___ counts, no more than ___ hours per sample per metal).

What one may like and what can reasonably be obtained is another thing. The number of counts one can get is totally dependent on the volume percent of that phase in the sample. The bulk concentration is a good indicator of this as well. If the bulk arsenic is 50 mg/kg I can tell you right now that I would have to spend days counting multiple splits (and adding them together) to get 200 counts. With elements like In, Tl Hg, and Se in these samples whatever I see will be all you get (0-10 particles if your lucky) This is why I setup the maximum time limit to be spent on one sample (8 hrs). After his amount of time its better to move on and just look at more samples.

5) We are proposing that additional documentation (electron micrographs) be included with the speciation results, in order to make the data more defensible. Please review this addition, and feel free to suggest alternate methods or changes. The following method for identifying when a photo will be taken is being considered:

- 2 photos per metal, per sample will be taken.
- grains to be photographed will be selected based on a random number generation, which will be supplied by ISSI when we give you the samples. So, for sample #____, we will provide a table that has grain # 2 and grain # 33 selected for photography.
- grains that were photographed should be recorded on the EMP graph (e.g., grain #2, #33), and documented in the Electron Micrograph Logbook (see attached excel file).
- additional photos should be taken at your discretion (anomalies, sites of interest, etc.), but should be kept separate, and labeled as opportunistic photos.

Any of these are fine with me!

6) We need the range of grain morphology (particle size, fraction, etc.) for each sample type: PAX, smelter soil and material, residential soil, etc.

OK

7) We are currently revising the SOP to incorporate these changes, and we would like for you to review it. This is a fast turn around, and we need your comments by 9 tomorrow (9/3) morning.

Thanks so much for your input on this, and please feel free to suggest any changes or additions to these proposed revisions. Please call (or email) Mary or myself if you have any questions: 303/292-4142 x 256.

EMP Standard Operating Conditions

	WDS	EDS
Accelerating Voltage	15 KV	15-20 KV
Beam Size	1-2 microns	1-2 microns
Cup Current	10-30 NanoAmps	10-30 NanoAmps
Ev/Channel	NA	10 or 20
Stage Tilt	NA	Fixed
Working Distance	NA	Fixed

MCA time Constant	NA	7.5-12 microseconds
X-ray lines **	S K-alpha PET	S K-alpha 2.31 KeV
	O K-alpha LDE1	O K-alpha 0.52 KeV
	C K-alpha LDEC	C K-alpha 0.28 KeV
	Zn K-alpha PET	Pb M-alpha 2.34 KeV
	As L-alpha TAP	Pb L-alpha 10.5 KeV
	Cu K-alpha LIF	Zn K-alpha 8.63 KeV
	Cd L-alpha PET	Cu K-alpha 8.04 KeV
	Pb M-alpha PET	As K-alpha 10.5 KeV
	Pb L-alpha LIF	As L-alpha 1.28 KeV
	In L-alpha PET	Cd L-alpha 3.13 KeV
Other Target Metals:	Tl L alpha LIF	In L-alpha 3.28 KeV
In	Hg L alpha LIF	Tl M-alpha 2.27 KeV
Tl	Se L alpha LIF	Tl L-alpha 10.26 KeV
Hg	Sb L alpha PET	Hg L-alpha 9.98 KeV
Se		Hg M-alpha 2.19 KeV
Sb		Se L-alpha 1.37 KeV
		Sb L-alpha 3.60 KeV

Table 8-2

Suggested Abbreviation for Photomicrographs

Target Metal	Metal-bearing Phase	Abbreviation
In	In	In
Tl	Tl	Tl
Hg	Hg	Hg
Se	Se	Se

Sb	Sb	Sb
	Lead Sulfide	Ga
	Lead Sulfate	Ang
	Lead Carbonate	Cer
	Mn-(M) Oxide	Mn(M)
	Fe-(M) Oxide	Fe(M)
	(M)Phosphate	(M)Phos
	Fe-(M) Sulfate	Fe(M)Sulf
	Metal Oxide	(M)O
	Pb-Mo Oxide	Wulf
	Slag	Slag
	Metallic Phase	(M)
	Metal Silicate	(M)Si
	Solder	Sold
	Paint	Pnt
	Metal-bearing Organic	(M)(Org)
	(M) barite	(M)Bar
	Pb arsenate	PbAsO
	Pb vanadate	PbVan
	As-Sb Oxide	AsSbO
	Chalcopyrite	Cp
	Sphalerite	Sph
	Arsenopyrite	Apy

Table 2-1

Metal-Bearing Forms Found Within Western Mining and Smelting Districts

OXIDES

Lead Oxide
Manganese (metal) oxide
Iron (metal) oxide
Lead molybdenum oxide

CARBONATES

Lead Carbonate
Zinc Carbonate

PHOSPHATES

Arsenic Oxide
Cadmium Oxide
Copper Oxides
Zinc Oxide
Lead Arsenate
Arsenic Trioxide
Calcium (metal oxide

SILICATES

Slag
Lead silicate
Arsenic silicate
Zinc silicate
Clays

SULFATES

Iron (metal) sulfate

Lead sulfate
Lead barite
Zinc Sulfate
Arsenic sulfate
Copper sulfate

(metal) phosphates

SULFIDES

Lead sulfide
Sulfur-containing salts
Iron-arsenic sulfide
Zinc sulfide
Copper sulfides
Copper-iron sulfide
Cadmium Sulfide

OTHER

Native: Lead,, Copper, Cadmium, Mercury, Indium, Thallium, Selenium

Lead/Arsenic/Cadmium/Mercury Chlorides
Lead paint
Solder
Organic lead
Lead vanadate

Minor telluride, and bismuth-lead phases